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**VAPOUR PHASE GROWTH OF THICK  
MONOCRYSTALLINE GaN EPITAXIAL LAYERS BY  
SANDWICH-METHOD.**

First Interim Technical Report

by

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13. ABSTRACT <b>We have modified a setup for growing GaN heteroepitaxial layers of large area and obtained 20 such structures on silicon carbide (SiC) and sapphire (Al<sub>2</sub>O<sub>3</sub>) substrates of 8 mm<sup>2</sup> in size 50-150 µm thickness by Sublimation Sandwich Method (SSM). The substrates have been analysed for optimal GaN layer growth conditions, using the available data, and the results indicated that hexagonal SiC (α-SiC) is the best material. The substrate/epilayer mismatches may be reduced by using Al-doped substrates pre-irradiated by high energy particles. We have studied the characteristics and structural quality of GaN layers deposited on SiC substrates, using X-ray diffractometry. The rocking curve measurements of GaN layers yielded the full width at the half maximum (FWHM) of 150 arc.sec. The manganese impurity was first observed and identified in GaN crystals grown by sublimation sandwich method by electron paramagnetic resonance.</b>				
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We have modified a setup for growing GaN epitaxial layers of large area and obtained 20 such structures on silicon carbide (SiC) and sapphire ( $\text{Al}_2\text{O}_3$ ) substrates of  $8\text{ mm}^2$  in size and 50–150  $\mu\text{m}$  thickness. The substrates have been analyzed for optimal GaN layer growth conditions, using the available data, and the results indicate that hexagonal SiC ( $\alpha$ -SiC) is the best material. The principal advantages of  $\alpha$ -SiC substrates are that they are insensitive to the GaN growth conditions and have the affinity of the lattice parameters ( $\Delta a/a=3.4\%$ ) and thermal expansion coefficients (difference in TEC=33%).

The substrate/ epilayer mismatches may be reduced by using Al-doped substrates pre-irradiated by high energy particles.

Our choice has been tested experimentally and has proved to be good for GaN layer growth. We have found that a sapphire substrate cracks when the layer thickness exceeds 150  $\mu\text{m}$ . In case of a silicon carbide substrate, cracking is observed only in the vicinity of the interface.

We have studied the characteristics and structural quality of GaN layers deposited on SiC substrates, using X-ray diffractometry. The rocking curve measurements of the GaN layers yielded the full width at the half maximum (FWHM) of 150 arc.sec. This value is comparable with the best FWHM of 96 arc.sec. for thin films (1  $\mu\text{m}$ ) reported by Nakamura and grown by the metal-organic deposition (MOCVD) technique.

Sometimes, the GaN epitaxial layers have a domain structure. The domains have the shape of a hexagonal prism of 200–300 nm in length and 50 nm along the surface.

The manganese impurity was first observed and identified in GaN crystals grown by sublimation sandwich method by electron paramagnetic resonance.

The results of this study have been reported in 3 papers (one of which was an invited talk) on 23rd International Symposium on Compound Semiconductors (ISCS-23) St.Petersburg, Russia, Sept. 23-27, 1996

#### List of reports

1. E.N.Mokhov and Yu.A.Vodakov Controlled growth of SiC and GaN by sublimation sandwich method (invited)// Programme and Summaries p.40
2. P.G.Baranov, I.V.Ilyin, E.N.Mokhov and A.D.Roenkov Investigation of manganese trace impurity in GaN crystals by electron paramagnetic resonance // item p.63.
3. M.E.Boyko Studying the temperature dependency of incommensurate domains in mercury halides and gallium nitride thick films // item p.63.

#### (2) Brief description of the research plans on staying period

1. Optimization of processing of a substrate surface before layers GaN deposition: drawing of a buffer layer, substrate etching.
2. Optimization of modes of growth of thick layers with the large area of a surface: of temperature modes; of geometry of modes systems; of conditions of submission of gas.
3. Characterization of GaN thick layers: structural perfection (x-ray method); optical, electrical and luminescent measurements; electron paramagnetic resonance study.

## Substrate Material Suitable for Thick GaN Layer Growth

### 1. Introduction

Group III nitrides are known to be promising materials for solid-state electronics, including short-wavelength devices and high temperature power and microwave devices [1,2]. Much progress has lately been made in the growth technology of group III nitrides, especially GaN. High quality GaN epilayers of hexagonal and cubic modifications are grown by MOCVD and MBE on sapphire and other substrates [1-4]. GaN layers of 0.5 mm thick are produced by hydride vapor-phase epitaxy [5].

There are however some serious problems involved in the growth of high quality thick epilayers and bulk crystals of these materials, because a larger thickness normally leads to a lower structural quality [5]. Besides, there are difficulties in controlling the doping levels of both bulk crystals and epilayers. The behavior of intrinsic defects in GaN is still poorly understood. As a result, the quality of such structures does not meet the current requirements on semiconductor materials.

We will first outline some specific features of these materials.

The materials do not melt but sublime on decomposition, making the growth from the melt impossible.

Equilibrium vapor pressure over the surface and the vapor phase composition are strongly non-stoichiometric. The vapor, unlike the solid phase, is rich in nitrogen; therefore, it is difficult to obtain material of homogeneous composition. Before the atoms align to form a crystal lattice, the molecules decompose into the respective elements, so there is a high probability that individual phases of group III elements will be formed on the surface of the growing crystal. This stimulates the formation of intrinsic nonequilibrium defects, e.g. vacancies, during the growth. The vapor phase of GaN is known to consist primarily of nitrogen molecules, so that nonequilibrium nitrogen pressure is very high, dozens and even hundreds of bars [6] at the growth temperatures.

To avoid GaN decomposition, the growth should be performed in a high pressure chamber, which is difficult but possible to implement [7]. Another common way used in available growth systems is to introduce activated nitrogen vapor, say, ammonia, into the chamber [8]. But in this case, the growth goes on under nonequilibrium conditions, since ammonia is unstable at the growth temperatures. The temperature rise is accompanied by an increase in nitrogen partial pressure over the GaN surface. For this reason, the growth temperature is usually made lower but then it becomes hard to achieve high growth rates due to a low surface mobility of GaN adatoms.

It has been shown that GaN can crystallize in various modifications, or polytypes, which can co-exist in the same growth conditions [9]. Such GaN polytypes as wurtzite and zincblende are known to differ in the stacking sequence of double layers of group III and V atoms. The physical properties of GaN polytypes also show a variation.

It is clear from this description that GaN and other III-V group nitrides are technologically challenging materials, and the growth of quality GaN crystals by CVD is possible only at relatively low growth rates ( $<10 \mu\text{m/h}$ ).

Earlier, we suggested an original method to control the epitaxial growth of high quality SiC [10, 11] and, later, GaN crystals [12] — the sublimation sandwich method, which can be applied to some other materials including decomposing compounds.

A simplified diagram of various sandwich growth cells is shown in Figure 1. The growth cell consists of a vapor source (1) and a single crystal substrate (2), on which a GaN crystal is grown. The source wafer is placed parallel to the seed crystal with a small gap. Ammonia can be introduced into the growth cell to prevent GaN decomposition. Growth conditions close to equilibrium can be created for a wide temperature range, from 1000 to 1300°C for GaN. Such a system may be quasi-closed for its vapors, leading to congruent transfer of the sublimated material from the source to the seed. As a result, we can grow bulk crystals and thick epilayers at a rate of 1 mm/h. The effective volume of the growth cell can be considerably reduced to obtain low levels of residual impurities and a high probability of utilizing all evaporated material, when its transport in the cell is close to 100%.

GaN layers were first grown by sandwich sublimation [12, 13] on silicon carbide and sapphire substrates in a horizontal quartz reactor with h.f. heating. GaN powder or metallic Ga were used as the source.

## 2. The Choice of Substrates for GaN Epilayer Growth

### 2.1 Influence of the Lattice Parameters

When choosing substrate material for growing single crystal epilayers, one should keep in mind the following factors:

- the crystal structure
- the lattice parameters
- the thermal expansion coefficient

It is important to know the elastic properties of both the substrate and the crystal to be grown, as well as the ductile-brittle transition temperatures. No doubt, the best substrates for epilayer growth are those made from the same material; this condition is known as homoepitaxy. Indeed, high quality epilayers and devices on their base have been produced using GaN substrates [14]. However, it is still difficult to produce perfect substrates, because the grown GaN single crystals have a large size and low structural quality.

Because of the lack of perfect substrates, of great importance is the problem of finding a suitable material for epilayer growth. It has been shown that a greater lattice mismatch of the layer and the substrate considerably deteriorates the characteristics of devices made from such structures.

The stress in a GaN heteroepilayer is reduced during the growth and cooling by introducing misfit dislocations on the interface.

For example, Monemar [15] found an appreciable difference in the exciton behavior in GaN epilayers grown on different substrates, which might be due to stress gradients in the layers. The importance of the stress effect was demonstrated by an appreciable change in the valence band dispersion in the layer plane and, hence, in the hole and exciton masses.

To grow a certain GaN modification, substrates of a similar crystallographic structure are preferable [2]. For example, cubic GaN layers can be grown on Si, GaAs, GaP and 3C-SiC substrates. Hexagonal GaN epilayers are, on the contrary, usually grown on sapphire, 6H-SiC and other substrates with the hexagonal structure. Much effort has so far been made to study wurtzite GaN because it is more stable than cubic GaN.

The characteristics of materials used as substrates can be found in Table 1.

Among other potential materials for substrates are spinel, NGO, TiO<sub>2</sub>, and LiAlO<sub>2</sub> [16].

Wurtzite GaN epilayers are usually grown on (0001) substrates, so we first focused on the lattice parameter mismatch along the a-axis ( $\Delta a/a$ ). These data are presented in Table 2. There is a data spread concerning the lattice parameter mismatch for the GaN/Al<sub>2</sub>O<sub>3</sub> system. Evidently, the most probable values are  $\Delta a/a = 14\text{--}16\%$  [17].

It is clear from Table 2 that ZnO, AlN, LiAlO<sub>2</sub>, and SiC have parameters similar to those of GaN. Nevertheless, GaN layers have so far been grown on sapphire substrates, because this material need not be pretreated or precleaned and is stable at high temperatures. But recently, the development of optimized buffer growth techniques has permitted one to minimize the effects of the lattice and thermal expansion mismatch to produce high quality MOVPE materials. GaN and AlN buffer layers, usually 300Å thick, are deposited at low temperature to produce a uniform coverage of amorphous material. Subsequent heating to the growth temperature of 1000°C transforms the buffer layer to a two-dimensional single crystal interface ready for epitaxial growth. Today, GaN layers grown by this method are the world record ones. There are however problems associated with residual stresses and defects producing a great lattice mismatch and differences in thermal expansion, the latter playing a major role in the post-growth cooling and annealing.

Good results have been obtained with sapphire substrates having a pre-deposited ZnO layer [5]. This approach has been shown to indicate the way for growing thick, high quality GaN layers.

Recently, interest in SiC substrates has considerably increased due to the progress in the growth of large bulk SiC crystals of 1–1.5 inch diameter and larger. Commercial production of such crystals has been started by the CREE company in the USA [18]. In this connection, we would like to emphasize that the sandwich method we have suggested allows the growth of not only thick epitaxial layers but also SiC bulk crystals (6H, 4H, and 15R) with a large diameter — up to 1 inch [19].

The lattice parameters have been shown to depend on the following major factors [20]:

- free electrons expanding the lattice in proportion with the strain potential of the conduction band minimum;
- the polytype structure;
- impurities and intrinsic defects (vacancies, antisites, interstitials), the difference in their ionic radii relative to the substitutional host ions (size effect), and stresses including those caused by the lattice mismatch of the substrate and the layer;
- linear defects (dislocations and clusters of point defects).

Free electrons change the lattice parameters as

$$(\Delta x/x)_{\text{electron}} = - Dn / (3B)n,$$

where  $x$  is the lattice constant,  $n$  is the electron concentration at the conduction band minimum,  $Dn$  is the strain potential of this minimum (–10eV for GaN), and  $B$  is the bulk modulus equal to  $2 \times 10^{11}$  Pa. This means that the free electron concentration of  $1 \times 10^{19}$  cm<sup>–3</sup> for GaN should raise the lattice constant approximately by 0.01%.

The lattice parameters for the various SiC polytypes can be found in [21]. They indicate a higher lattice parameter  $c$  and a lower parameter  $a$ , as the proportion of hexagonal SiC becomes larger. The lattice parameter variation does not exceed 0.1% for various polytypes.

Later, we will discuss in more detail the effect of impurities and defects on the SiC lattice parameters with emphasis on those leading to their higher values, since the lattice constant of SiC is smaller than that of GaN.

One example of such an impurity is aluminum substituting silicon in the SiC lattice. The tetrahedral radius of an Al atom ( $r_{\text{Al}}=1.26\text{\AA}$ ) is larger than that of Si ( $r_{\text{Si}}=1.17\text{\AA}$ ). Besides, Al possesses a high solubility level in SiC ( $C_{\text{Al}}=2\times 10^{21}\text{ cm}^{-3}$ ) [22], leading to a larger lattice constant, but this gain is only 0.01-0.02% in heavily Al-doped SiC crystals [23].

The formation of intrinsic nonequilibrium defects, say, during the crystal irradiation by high energy particles (ions, fast neutrons, electrons) also increases the lattice parameters, e.g. they increase by 1% after  $10^{21}\text{ cm}^{-2}$  neutron irradiation [24]. Nonequilibrium defects have been shown to be stable up to 1300–1400°C. This temperature is higher than the GaN epilayer growth temperature.

Nonequilibrium defects produced during the crystal irradiation or growth may form stable clusters, vacancies and interstitials, which affect the mechanical and other properties of the crystals. Irradiated SiC crystals with nonstoichiometric defects tend to cracking, which results in deterioration of their microstrength characteristics.

The lattice mismatch of the substrate-epilayer system is also affected by the substrate orientation. It is interesting that different mismatch values can be observed for the polar (0001) faces. This fact was interpreted in [26] as being due to the contribution of the interface charge from the ionic portion of the binding energy. Estimation of the lattice mismatch at the GaN-SiC interface has given the following values: 3.4% for both assumingly uncharged GaN/SiC interfaces, 2.6% for the charged (0001)-Si interface, and 5.6% for the charged C interface.

This assumption has been used to explain the essential difference in the surface structure of GaN layers grown on the (0001) C and (0001) Si sides of a SiC substrate [27]. Indeed, GaN layers grown on C-terminated (0001) SiC substrates exhibit a pyramidal structure, which may be accounted for by a considerable local micromismatch. On the other hand, GaN layers grown a Si surface are smooth because of a smaller mismatch on this surface. Therefore, the Si surface is preferable to the C surface for the growth of GaN layers on a (0001) SiC substrate.

## 2.2 Effect of the Thermal Expansion Coefficient

Thermal expansion of single wurtzite GaN crystals has been studied in the temperature range from 300 to 900 K or from 80 to 820 K. Maruska and Tietjen [28] reported a linear temperature variation of the lattice constant with the thermal expansion coefficient  $+5.59\times 10^{-6}\text{ K}^{-1}$ . The gain in the lattice constant  $c$  shows a superlinear temperature dependence. The average coefficient of thermal expansion along the  $c$ -axis is  $+3.17\times 10^{-6}\text{ K}^{-1}$  at  $T + 700\text{--}900\text{ K}$ .

Sheleg and Savastenko [29] reported the thermal expansion coefficient to be  $+4.52+0.05\times 10^{-6}$  and  $+5.25+0.05\times 10^{-6}$  at 600 K for the normal and parallel directions relative to the  $c$ -axis, respectively.

Thermal expansion in GaN has recently been studied in [30]. Two kinds of samples were used: bulk samples and GaN epilayers grown on sapphire substrates. The latter had a smaller lattice constant, by about 0.03%, in the direction parallel to the interface plane. This difference was due to a greater thermal expansion of sapphire relative to the GaN layer.

The thermal expansion coefficient is shown to depend on many parameters, such as defect content, free-charge concentration, and stress [5]. This is likely to account for the considerable dispersion of experimental GaN thermal expansion data presented in



Figure 2. The difference in the linear expansion coefficients of the substrate and the epilayer gives rise to structural defects near the interface during the cooling to room temperature. This is especially dangerous at relatively low temperatures when the crystal loses its plasticity. The stresses produce cracks in the substrate and epilayer. The temperature range for the ductile-brittle transition is found to be 800–1100°C for SiC [30]. The respective range for GaN is not known exactly. However, the thermal stress due to different expansion coefficients of GaN and sapphire has been shown to arise mainly at temperatures below 500–600 K [31]; above this threshold, the lattice mismatch is close to zero.

Our experience shows that the relative deformation ( $\Delta L/L$ ) in 6H-SiC crystals larger than  $10^{-3}$  at room temperature is critical and leads to the crystal damage. Assuming that the plasticity of GaN is not smaller than that of SiC, we consider this value as being critical. Therefore, the difference in the expansion coefficients of the substrate and the GaN epilayer should not lead to deformation in GaN crystals larger than  $10^{-3}$  on cooling from 800°C to room temperature.

Some expansion coefficients for materials used in GaN heteroepitaxy are presented in Table 1. Their temperature dependence for GaN, SiC and sapphire is shown in Figure 1. These data can be used to calculate the expansion differences between GaN and the substrate materials, which vary from -25% for sapphire to +33% for silicon carbide. Note that this difference for  $\text{LiAlO}_2$  is -21% [16].

Thus, the available data and our own estimations of the lattice parameters and expansion coefficients for GaN and various substrate materials over the temperature range from 300 to 800°C indicate that hexagonal silicon carbide is preferable as substrate material for GaN heteroepitaxy.

This conclusion has been supported by the results of a study [32] on the residual stress in GaN epilayers grown on sapphire and 6H SiC substrates. In both cases, a large difference in the stress relaxation was observed. GaN layers on SiC substrates are subject to tensile stress and experiences a nearly complete relaxation. On the contrary, there is an appreciable residual stress in GaN epilayers on sapphire, and the stress conditions also differ. This could be attributed to a different signs of lattice mismatches and thermal expansion coefficients of sapphire, SiC and GaN, and SiC that reduce the epilayer strain during the post-growth crystal cooling.

Moreover, SiC crystals may have any polarity and conductivity (both n- and p-type) with a wide range of resistance, from  $10^{-2}$  to  $10^8 \Omega\text{cm}$ .

### 3. Conclusion

SiC substrates are preferable for heteroepitaxial growth of GaN layers of large area and thickness. Such substrates are well matched with the GaN layer in both the lattice parameter and the thermal expansion coefficient. The GaN/SiC lattice mismatch can be reduced by using Al-doped 8H-SiC irradiated by high energy particles. Various manifestations of stress due to the lattice and expansion mismatch in SiC and GaN reduce the epilayer strain during the post-growth crystal cooling.

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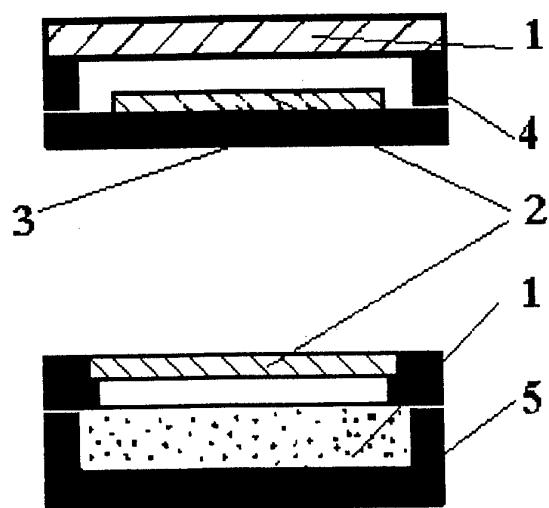


Fig.1. Two types of sandwich cells

1- source; 2 - substrate ; 3 - plate;  
4-ring; 5 - cup

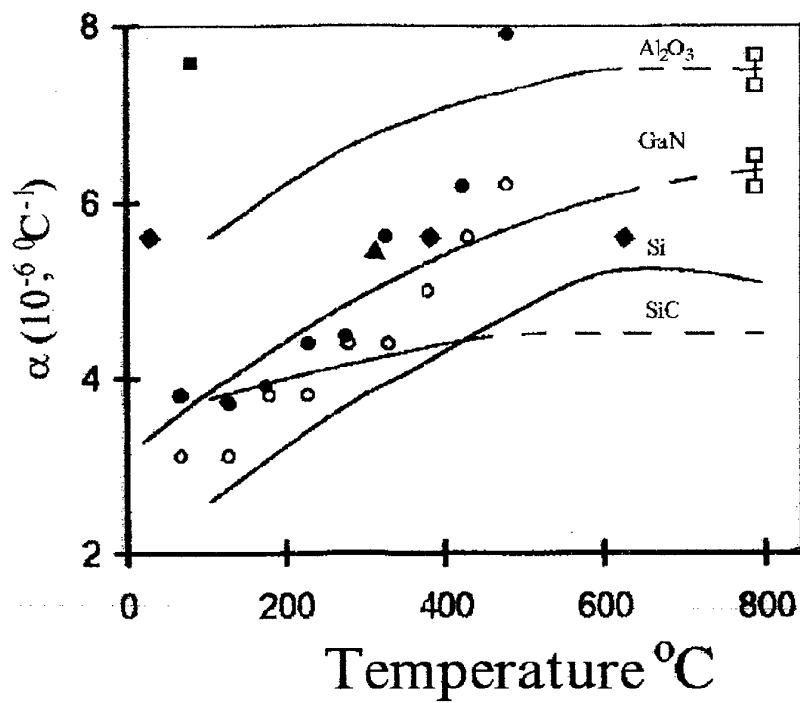


Fig.2. Dependence of thermal expansion coefficients of SiC, Si, GaN and  $\text{Al}_2\text{O}_3$  on temperature

Data of Si, SiC and  $\text{Al}_2\text{O}_3$  from book: Fizicheskie velichini.

Ed. by E.M.Voronkova et al, Moskow, 1991

Experimental points for GaN from [28] and [30] and our evaluations.

**Table 1****Properties of prospective nitride substrates.**

Substrate material	Symmetry	Lattice parameters Å	Coefficient of thermal expansion $\times 10^{-6}/\text{K}$
GaN-WZ	Hexagonal	a=3.189 c=5.185	5.59
GaN-ZB	Cubic	a=4.503	3.17
AlN	Hexagonal	a=3.112 c=4.9825	4.2 5.3
6H SiC	Hexagonal	a=3.08 c=15.12	4.2 4.68
Sapphire	Hexagonal	a=4.758 c=12.99	7.5 8.5
ZnO	Hexagonal	a=3.252 c=5.213	2.9 4.75
GaAs	cubic	a=5.6533	6.0
3C-SiC	cubic	a=4.36	
MgO	cubic	a=4.216	10.5
InP	cubic	a=5.8693	4.5
GaP	cubic	a=5.4512	4.65
MgAl <sub>2</sub> O <sub>4</sub>	cubic	a=8.083	7.45

The data from Landolt - Bornstein (Springer, New York, 1982), V.17

**Table 2****Substrate Materials for GaN**

Material	Structure	% Mismatch, ( $\Delta a/a$ )
AlN	Wurtsite	2.4
Sapphire	Wurtsite	8.3; 13.8*; 16**
GaAs	Zinc blende	9.0
GaP	Zinc blende	5.2
Si	Cubic	4.8
6HSiC	Hexagonal	3.4
3C SiC	Zinc blende	3.15
SiO <sub>2</sub>		4.05
LiAl <sub>2</sub> O <sub>4</sub>	Wurtsite	1.45**
ZnO	Wurtsite	0.2

The data from [8]; \*- from [26]; \*\* - from[16].